

ABSTRACT for 28th Annual Conference on Composites, Materials and Structures
(U.S. Only/ITAR-Restricted Sessions)
January 26-30, 2004 Radisson Resort at the Port, Cocoa Beach/Cape Canaveral, Florida

Goals and Progress for Mil Handbook 17, Vol. 5 on Ceramic Matrix Composites- Testing and Characterization

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The initial edition of the Mil Handbook 17, Vol. 5 on ceramic matrix composites (CMCs) was recently published. Part C of this CMC volume describes procedures for characterization of the thermal, physical, and mechanical properties of CMCs and subsequent data analysis procedures. As is the case for entire CMC volume, this chapter is a "work in progress" with sections on recommended test plans and matrices, data reduction, and test methods. This presentation will describe the current status in development of this section of the CMC volume and will highlight examples of critical issues that will be addressed in this volume. CMC systems are a different class of material than metals or polymer matrix composite with different mechanical response and failure modes, design rules, test methods, and characterization techniques. The CMC test protocols need to include guidance on how to generate enough reliable data in order to estimate, with reasonable confidence, the lower bounds of performance properties, such as strength and life. Strength data sets on two material systems, C/SiC and Nicalon/SiNC, and a creep rupture data set on C/SiC, will be presented to highlight this issue. The structural properties of CMC parts can sometimes be significantly different than those obtained from flat panels because of changes in fiber architecture often required to make shapes and spatial and lot-to-lot variations in processing conditions. To illustrate an example, the hoop tensile properties of SiC/SiC cylinders will be compared to properties obtained from flat panels fabricated from the same composites. These CMC testing-design issues are being defined and documented in the Mil Handbook 17 CMC volume.



Mil Handbook 17 Vol 5

Ceramic Matrix Composites

Testing and Characterization Section: Goals and Progress

January 28, 2004
Cape Canaveral, FL

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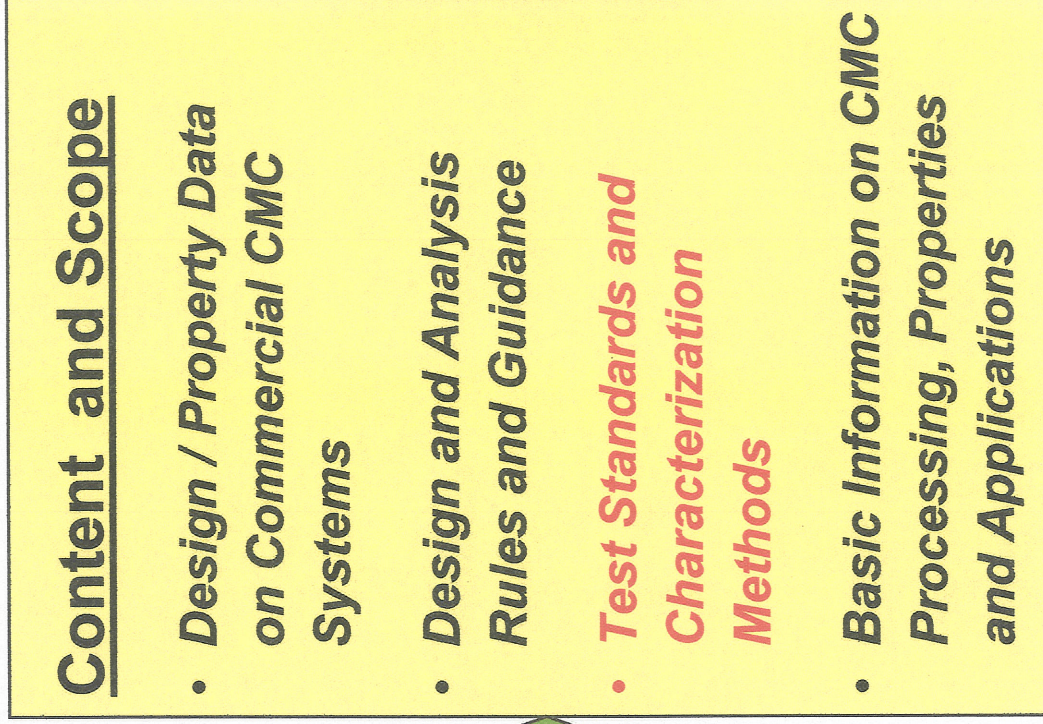


Presentation Objectives



1. Give a brief description of the Mil 17 Handbook on CMCs.
2. Educate everyone on the existing CMC testing and characterization section.
3. Discuss examples of issues that still need be addressed in the testing section of Mil 17 Handbook:
 - a) How many repeat tests are required to obtain strength and durability properties with statistical confidence?
 - i. Strength and life data sets for C/SiC and strength data set for Nicalon/SiNC will be presented.
 - b) How representative is CMC panel data to CMC component properties?
 - i. Hoop tensile properties of SiC/SiC cylinders will be compared to panel tensile properties for same materials.
4. Ask for volunteers.

Scope and Approach of Handbook



Input Approach

- Validated, Statistically Based Property Data
- Work with CMC Suppliers and CMC Designers

Output Approach

- Today -- Paper and PDF Files on CD RM
- Tomorrow -- Interactive Database on the WEB

Vision Statement

- To be the primary and authoritative source for recommended/required methods and matrices for testing and characterization of CMCs and their constituent materials.

Goals:

- To identify appropriate existing consensus standard test methods for CMCs and their constituent materials and to assist in the development of appropriate standard test methods for CMCs and their constituent materials, where no such standards exist.
- To develop test programs required for preliminary design of a CMC component.
- To recommend statistically-based test matrices and subcomponent test procedures and plans necessary for final design of a CMC component.

Current CMC Systems in the Database

<u>Composite Name</u>	<u>Composite Description</u>	<u>Producer</u>
• 9/99 EPM SiC/SiC	Sylramic SiC fiber/BN-SiC/MI SiC	GE Power Systems
• Enhanced SiC/SiC	SiC/Carbon/SiC	GE Power Systems
• Carbon/SiC	T300 Carbon/Carbon/CVI SiC	GE Power Systems
• Hi-Nicalon/MI SiC	SiC/BN/MI SiC	GE Power Systems
• AS-N720-1	Oxide/Oxide	COI Ceramics
• Sylramic S-200	SiC/BN/SiNC	COI Ceramics

The Handbook always welcomes:

- Additional data on materials already in the database.
- *Durability data is lacking.*
- Data on other CMC systems
- *Data from other composite producers is desired.*

Outline of Section on Characterization and Testing

Characterization &

Testing

- ✓ Test program planning
- ✓ Material screening
- ✓ Material qualification
- ✓ Review of national/international standards for CMC thermal, mechanical and physical testing
- ✓ Lessons Learned

Next Phase

- * Durability Testing
- * Failure Analysis
- * Microstructural and Chemical characterization
- * Environmental testing
- * Subcomponent testing



Details of Existing Outline of Testing and Characterization Section



8 Chapters (of 18 Total for CMC Handbook)

Chapter 8 -- Testing Overview

Chapter 9 -- Material Testing for Data Submission

Chapter 10 -- Reinforcement Evaluation (no text)

Chapter 11 -- Matrix Evaluation (no text)

Chapter 12 -- Interface Evaluation (no text)

Chapter 13 -- Composite Evaluation (contains existing test standards)

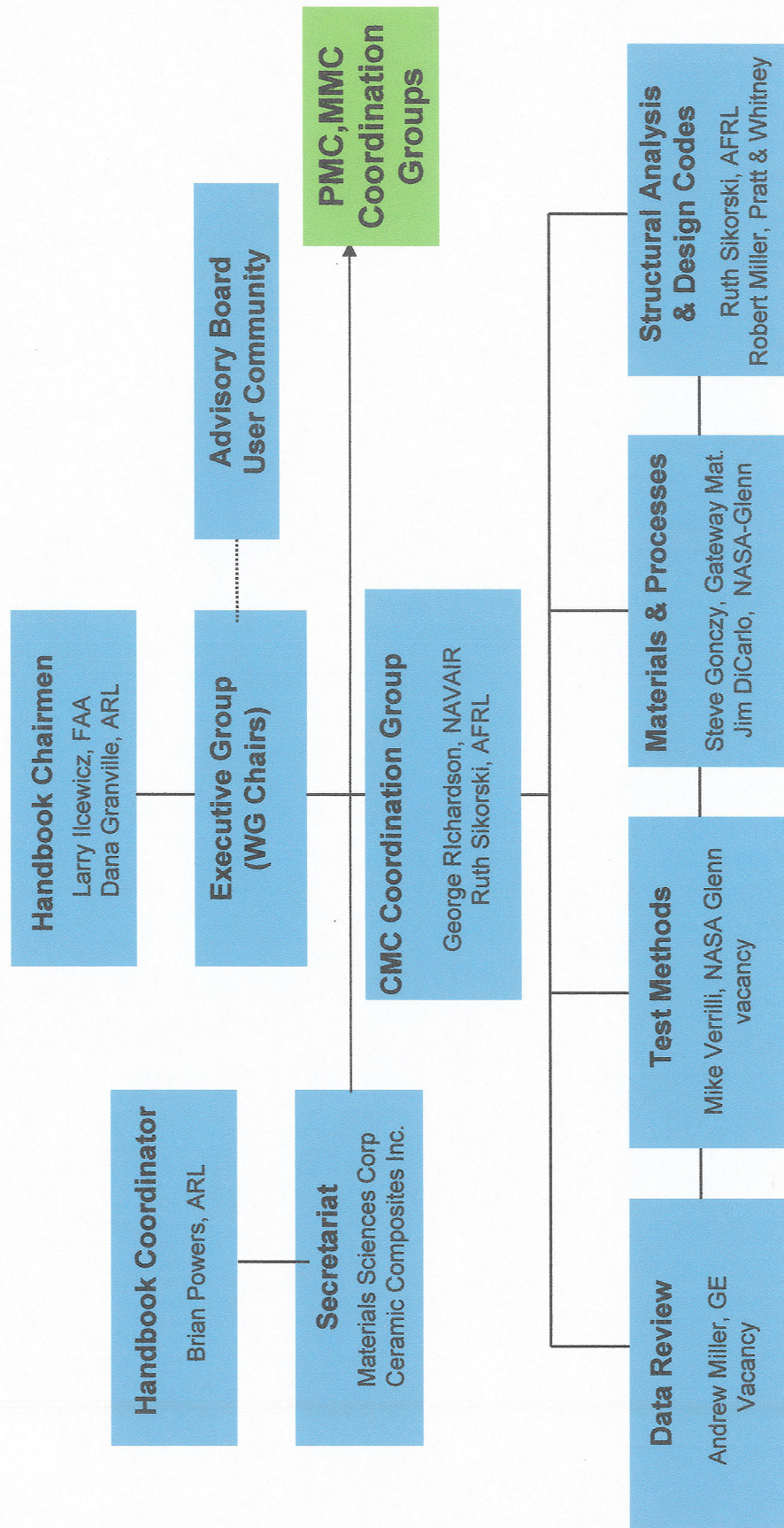
Chapter 14 -- Subcomponent Testing (no text)

Chapter 15 -- Machining and Grinding (no text)

Chapter 16 -- Statistical Methods (text from PMC book)

- Current organization of this section is under evaluation.

Mil Handbook 17 Working Organization





Examples of Testing Issues that still need be addressed in Mil 17 Handbook



- Testing of CMC hoops
- Strength distributions of a CMC versus test temperature.
- CMC material variability

Hoop Tensile Characterization of SiC/SiC Cylinders Fabricated from 2D Fabric

Background

Annular combustor liner operation requires CMC material to resist thermally-induced circumferential (hoop) and axial bending stresses.

Structural properties of CMC's in components can be different that those measured from CMC panels.

Objective

Measure hoop tensile properties of SiC/SiC cylinders.

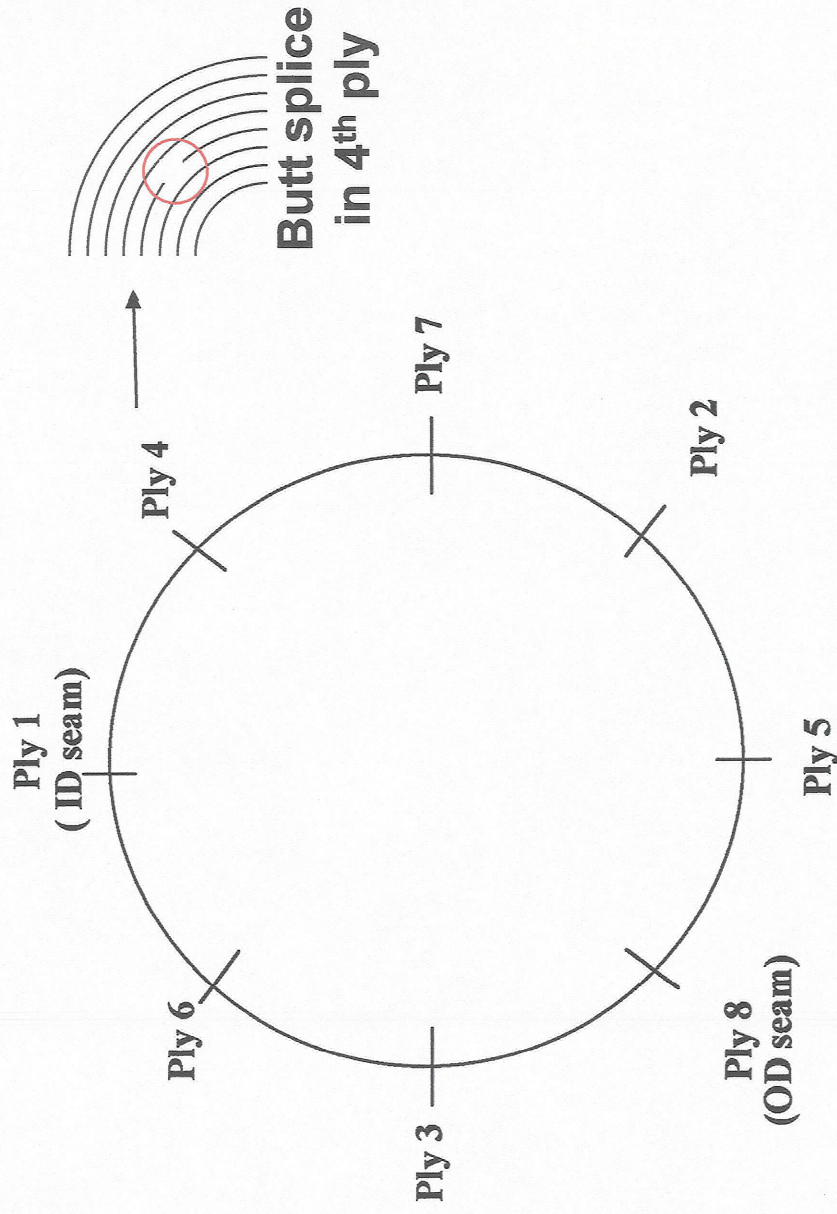
Cylinders of several material variants were tested.

Tests conducted at 25 and 1200°C.

Compare hoop and panel tensile behavior.

Does data from panels represent hoop properties from cylinders?

2D Ply Lay-Up for 4" Diameter SiC/SiC Cylinders Reinforced with [0/90] 5 Harness Satin SiC cloth (8 plies)

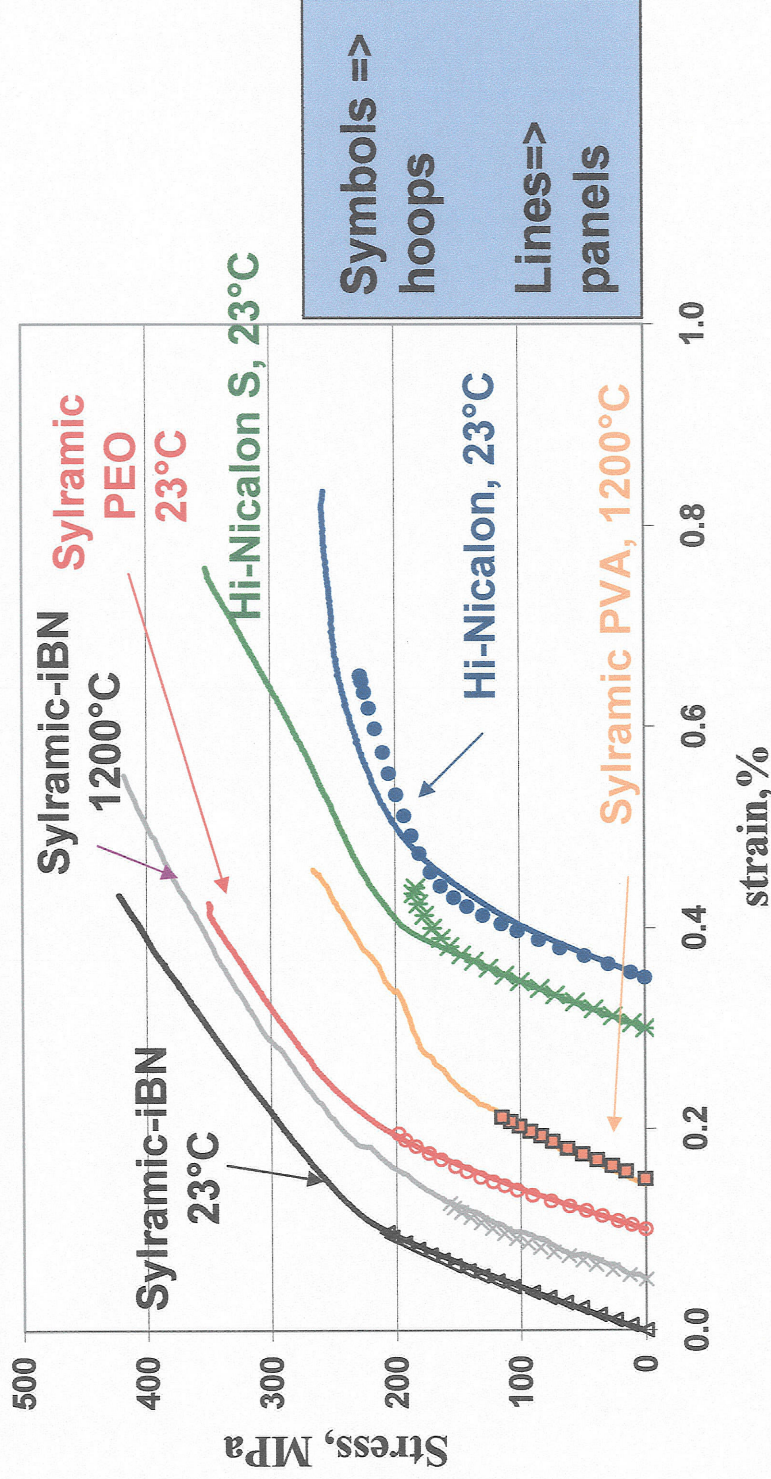


- Example is for an 8 ply cylinder.
- Each ply seam is separated by 45°.
- Adjacent ply seams are 135° apart.

- Butt joints used for cylinder ply seams.
- Ply seams are necessary for annular parts with non uniform cross-sections that are fabricated from 2D cloth.

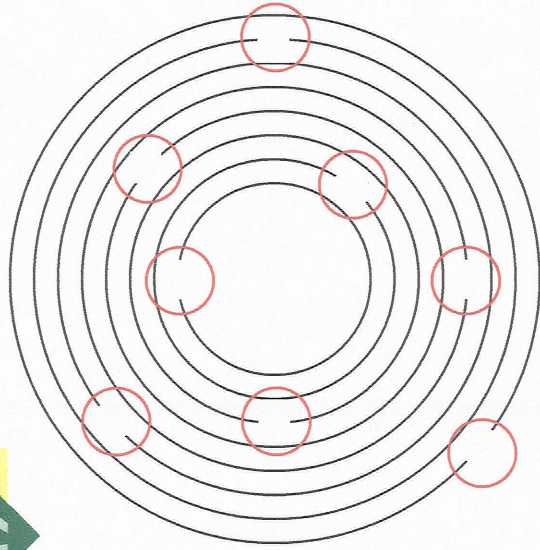
Comparison of Stress-Strain Behavior of SiC/SiC Cylinders and Panels with a CVI+MI SiC/Si Matrix

5 different fibers used to make cylinders and panels



- Room temperature and 1200 °C data are shown.
 - Initial panel and hoop stress-strain behavior is the same.
 - Hoop strengths are at most 50 % of panel strengths.
- Exception was the cylinder with Hi Nicalon fibers.*

Why Are Tensile Strengths and Failure Strains of SiC/SiC Hoops Much Lower than Panel Properties?



Schematic of hoop cloth layout for an 8 ply cylinder showing locations of butt splices

Fiber volume fraction at each butt splices is lower than rest of composite.

An 8 ply cylinder would have 14 % less fibers at the plane of the butt splice.

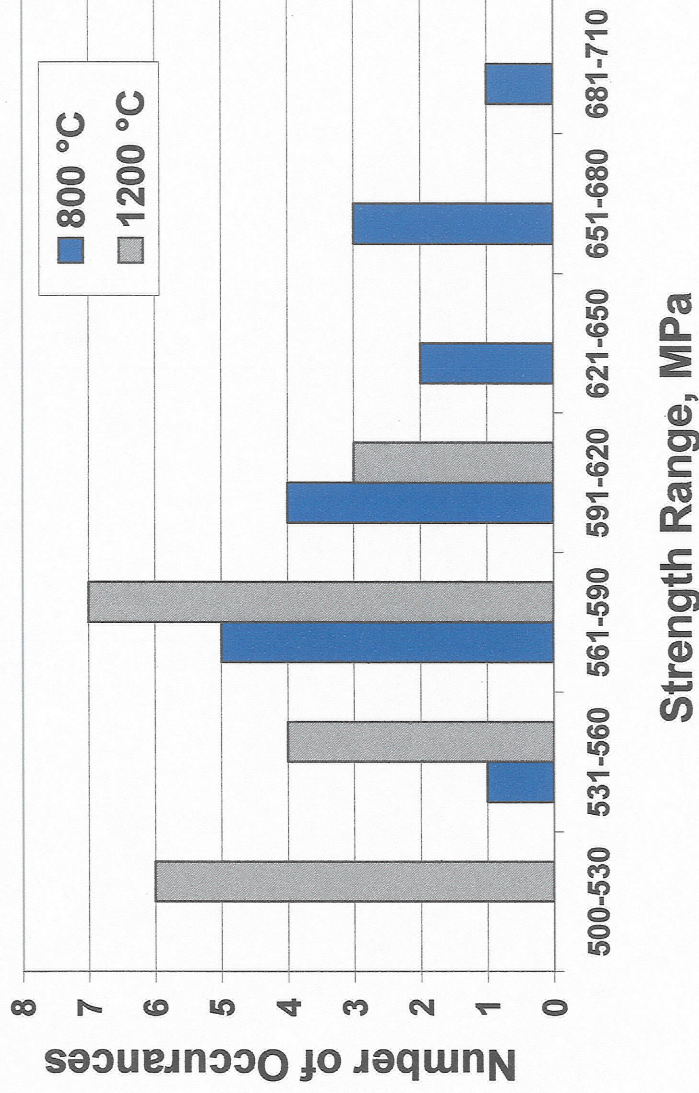
However, hoop strengths were 50% of panel strengths.

- Microstructural examination revealed that fracture of hoops occurred at ply splices and fracture always occurred at the inner ply splice.
- This suggests *that stress concentration effects exist at butt splices.*
- The Mil 17 testing section will include a section on subelement testing.

Comparison of Tensile Strength of [0/90] C/SiC specimens at 800 and 1200 °C



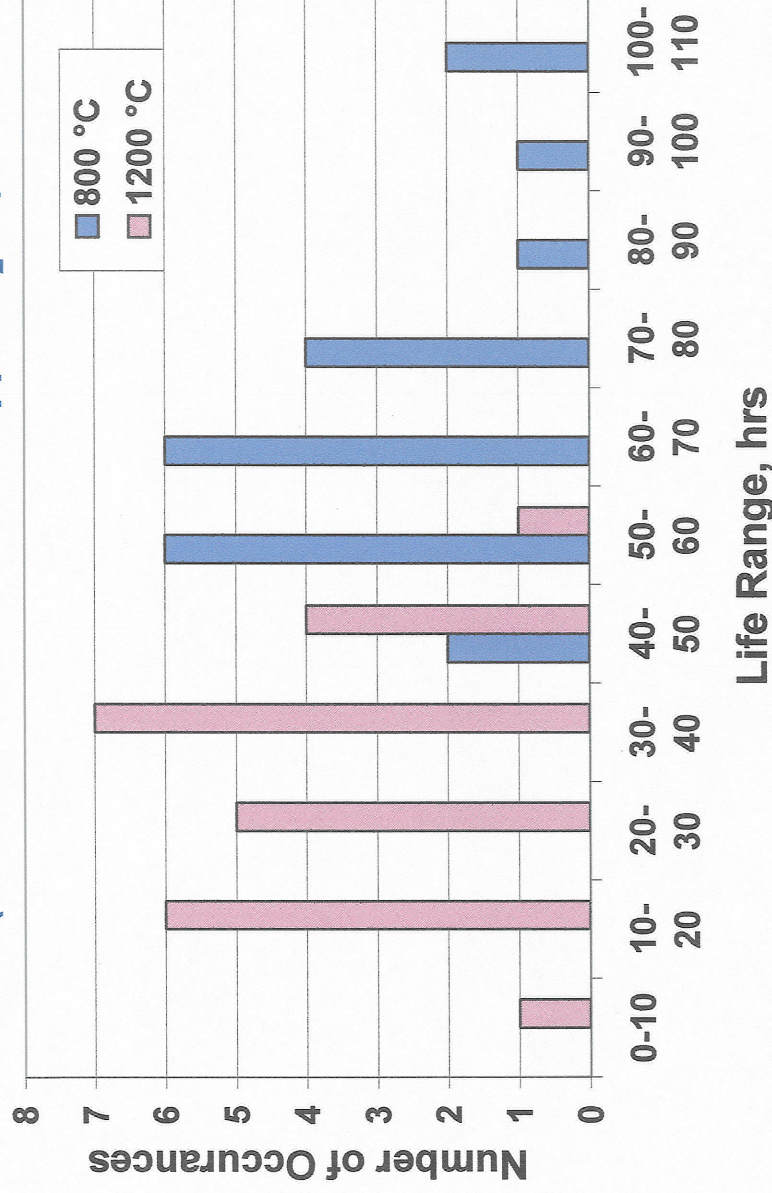
(test environment = 1000 ppm O₂/Ar)



- Average strength (from 20 tests at each temperature):
800 °C: 609.9 ± 37.4 MPa and 1200 °C: 558.1 ± 29.6 MPa.
- All tests conducted on 1 lot of C/SiC.
- Mil 17 handbook needs to include information on proper statistical methods to analyze CMC strength data.

800 and 1200 °C Rupture Life Data for [0/90] C/SiC Specimens (all tested at 207 MPa)

(test environment = 1000 ppm O₂/Ar)



- Average rupture life (from 20 tests at each temperature):
800 °C: 69.0 ±17.22 hrs, 1200 °C: 28.84 ±11.72 hrs.
- Are additional data needed to obtain a reliable standard deviation?
- Do these observations apply to other CMC's?

Round Robin Tensile Testing of Nicalon/SiNC

OBJECTIVES

- Determine precision of procedures in ASTM C1275 tensile test method for CMC's.
- Evaluate the robustness of the test method against variations in test procedures.
- Determine statistical distribution of tensile properties for a CMC material.

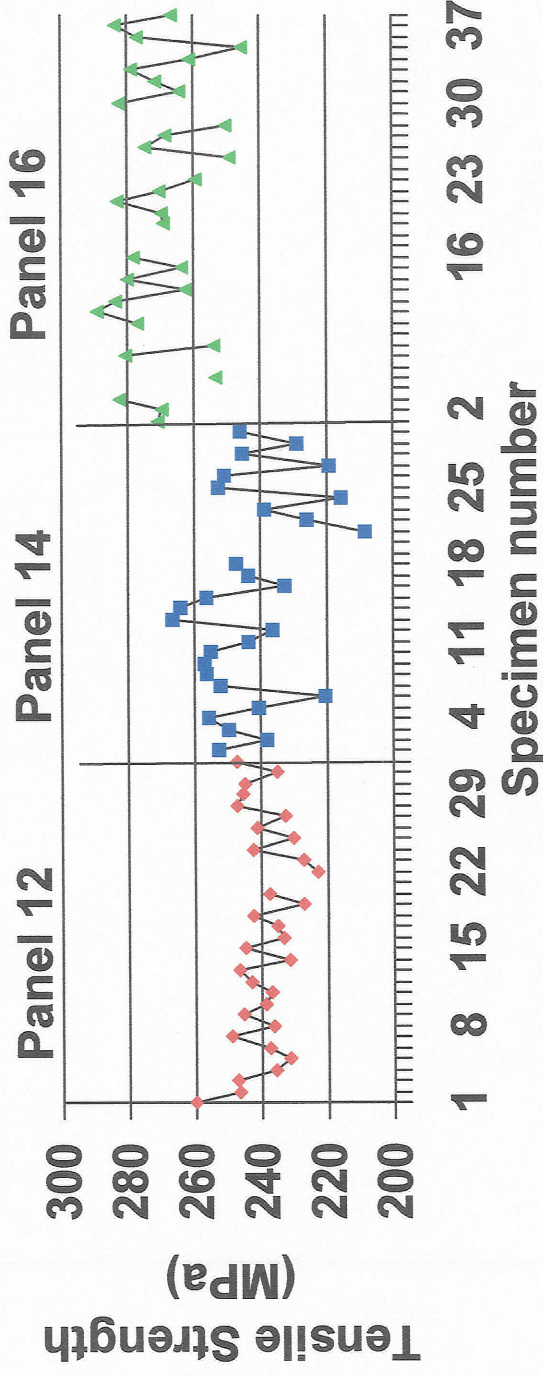
APPROACH

- Material: 8-ply Nicalon™ (8HS) Sylramic™ S-200 Composites
- Ambient temperature tensile testing
- Nine sets of 10 tensile bars tested at 9 laboratories per ASTM C1275 - Tensile Test Method for CMCs

Reference: M. Jenkins and L. Zawada, "Detailed Study of the Tensile Behavior of a 2D Nicalon/Sylramic CMC", ASTM STP 1392, 2000, pp.48-61.

Tensile Strength Measured 3 Nicalon/SiNC Plates

Data for 90 specimens from 3 panels, randomly selected and tested at 9 labs



- ANOVA analyses confirmed statistical difference in mean strengths obtained from the 3 panels.

Panel 16 specimens had the greatest mean strength.

Same trend found in the tensile failure strain data.

- The modulus and PL strengths of the three panels were similar.
- *Mil 17 Handbook needs this type of data to aid in development of recommended test matrices.*

Test Procedure Variations Observed in Results of Round Robin Tensile Testing of Nicalon/SiNC

Statistical Effect (t-test at 95% Confidence) on Strength Strain Modulus

Drive Mechanism -- 4 laboratories used screw drive machines; 5 laboratories used hydraulic systems. None 3% Δ

Pre- and Post-Test Bending -- Measured values were all less than 5%, with a low of 0.2% and a high of 4.9% None None

Grip Pressure -- Grip pressures ranged from 0.2 MPa to 8 MPa None None

Load Train Couplers -- 2 laboratories used self-aligning fixtures; 9 used rigid None None

Measured humidity -- From 34% RH to 62% RH. None None

Extensometer Gage -- 1 lab used a 12.5 mm gage; 7 used a 25-33 mm gage length ? ?

- There were variations in tensile means between labs but no test method variable had a dominant effect.
- Mil 17 testing section includes references to existing test standards.



SUMMARY

- **Mil Handbook 17 on CMC's is a work in progress.**

- Considerable effort and expertise are required to complete the Testing and Characterization section.
- We need your participation and support.
 - *Review and comment on draft sections*
 - *Write and edit new sections.*
 - *Provide additional data on CMC materials.*
 - *A short informational meeting will be held here at 5:30 today.*

- **Next Working Meeting will be held in May 2004.**

- To be held on West Coast, location TBD.
- For further information, contact:

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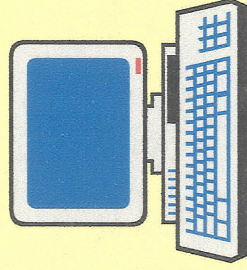


For More Information on Mil 17



Check the Mil Handbook 17 Website

www.mil17.org



or Contact the Secretariat --

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